WATER UNSTABLE FOAM

Technical field of the invention

The present invention relates to foam components, typically particles, comprising a matrix formed from a polymeric material and a plasticiser, a dissolution agent and an active ingredient, such as a detergent active ingredient, typically to be delivered to an aqueous environment.

Background to the invention

Compositions such as cleaning products and personal care products, cosmetic products and pharmaceutical products, often comprise active ingredients which are to be delivered to water or which are required to be active in an aqueous environment. Many of these active ingredients are sensitive to moisture, temperature changes, light and/or air during storage.

Another problem with many of these active ingredients, in particular enzymes, is that they tend to form dust due to physical forces directed upon them during handling. This not only creates waste product, but the dust can also cause hygiene and health problems.

Attempts to overcome these problems have led to the development of protecting these active ingredients by coating agents or encapsulating agents. The problem with many of these coated particles is that they do not always exhibit sufficient impact resistance during handling, and when acted upon by physical forces typically encountered during handling, dust is formed which can cause hygiene and health problems. Also, these coated particles are not always readily soluble in aqueous environments and exhibit poor dissolution properties upon contact with water.

The Inventors have found an improved method of protecting active ingredients and delivering these active ingredients to aqueous environments. They have found that

specific foam components comprising a matrix formed from a polymeric material and a plasticiser, are very impact robust and the active ingredient which is incorporated therein is protected against physical forces acting upon said foam component. Furthermore, the Inventors have found that when a dissolution aid is also incorporated in the foam component, the foam component dissolves or disintegrates readily upon contact with water releasing the active ingredient to the aqueous environment.

Thus, the foam component of the present invention is very impact resistant, thus resulting in reduced breaking-up or abrasion during handling and reduced dust formation, and readily soluble upon contact with water. For example, foam components, such as particles or beads, comprising enzymes can be obtained which are safer and more efficient to handle and use. Moreover, these component can be made such that they deliver the active ingredients incorporated therein, such as enzymes, very efficiently to an aqueous environment. The component of the present invention is air-stable under normal humidity storage conditions, but unstable upon contact with water, to thus deliver the active ingredient. The foam component is useful in any product, especially useful in cleaning products, pharmaceutical products, personal care products, cosmetic products and fabric care products.

Summary of the invention

The present invention provides a foam component comprising a mixture of a polymeric material, a dissolution aid, and an active ingredient, preferably being active in an aqueous environment, the foam component being stable upon contact with air and unstable upon contact with water.

The dissolution aid improves the water-solubility or water-disintegration of the foam component.

It may be preferred that the dissolution aid of said foam component comprises an effervescence system, a hydrotrope, or a combination thereof.

The present invention also relates to processes for making the foam component. Preferably the foam component is obtainable by a process comprising the steps of;

a) obtaining a polymeric material; and

b) chemically or physically introducing a gas in said polymeric material; and

c) prior to step b) and/or simultaneous with step b) and /or subsequent to step b), contacting an active ingredient to said polymeric material; and

d) prior to step b) and/or simultaneous with step b) and /or subsequent to step b), contacting a dissolution aid to said polymeric material; and

e) shaping the components of the resulting polymeric material;

whereby preferably one or more steps a) to e) are followed or accompanied by the removal of part of the water, if present.

In another embodiment of the present invention, the use of a foam component is provided, to deliver active ingredients to an aqueous environment, preferably the active ingredients being detergent active ingredients, preferably enzymes, and the aqueous environment being the wash water.

Detailed description of the invention

Foam component

The foam component of the present invention, herein referred to as "component", comprises an active ingredient, a matrix and a dissolution aid. Said active ingredient, matrix and dissolution aid are described in more detail hereinafter.

Said component herein is preferably water-dispersible, water-disintegrating or water-soluble. Preferred water-dispersible components herein have a dispersibility of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out hereinafter using a glass-filter with a maximum pore size of 50 microns; more preferably the component herein is water-soluble or water-disintegrating and has a solubility or

disintegration of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out hereinafter using a glass-filter with a maximum pore size of 20 microns, namely:

Gravimetric method for determining water-solubility, water-disintegration or water-dispersibility of the component herein:

50 grams ± 0.1 gram of the component herein is added in a 400 ml beaker, whereof the weight has been determined, and 245ml ± 1ml of distilled water is added. This is stirred vigorously on magnetic stirrer set at 600 rpm, for 30 minutes. Then, the component - mixture is filtered through a folded qualitative sintered-glass filter with the pore sizes as defined above (max. 20 or 50 microns). The water is dried off from the collected filtrate by any conventional method, and the weight of the remaining component fraction is determined (which is the dissolved, disintegrated or dispersed fraction). Then, the % solubility, disintegration or dispersibility can be calculated.

The component herein is typically used to deliver actives to aqueous environment. Then, the component herein, and preferably the matrix thereof, is unstable when brought into contact with water. This occurs such that the active ingredient(s) or part thereof, present in the component is delivered to a liquid, preferably an aqueous environment such as water. Preferably the component or part thereof denatures, disintegrates, preferably disperses or dissolves in liquid, preferably in an aqueous environment, more preferably in water. It may be preferred that the active ingredient is delivered rapidly to water and that the component is such that it disperses or dissolves rapidly; preferably at least 10% of the component, by weight, is dissolved or dispersed in 30 minutes after contacting said component with water, or more preferably at least 30% or even at least 50% or even at least 70% or even at least 90% (introduced in the water at a 1% by weight concentration). It may even be preferred that this happens within 20 minutes or even 10 minutes or even 5 minutes after contacting the component with the water. The dissolution or dispersion can be measured by the method described hereinbefore for measuring the dissolution, disintegration and dispersion of the component herein.

Preferably the component is such that the total volume of the component is changed, preferably reduced, with at least 10%, compared to the initial total volume, as for example can be determined when 1 cm^3 of the component is added to100 ml of demineralised water upon and stirred for 5 minutes at a speed of 200rpm, at a temperature of 25°C. Preferably the change, or preferably reduction, in total volume is at least 20% or even at least 40% or even at least 60% or even at least 90% or even about 100%, e.g. because it may be preferred that substantially the whole component is disintegrated, dispersed or preferably dissolved into the water quickly.

This can be measured by use of any method known in the art, in particular herein with a method as follows (double immersion technique):
1 cm^3 of a component is obtained and introduced in a 100 ml micro volumetric measuring cylinder which is filled with 50 ml ± 0.1ml of an organic inert solvent. Acetone is for example used when found to be neither denaturing and/or not interacting with the polymeric material in the matrix of the component herein, for example when this is PVA. Other neutral organic medium can be used according to the nature of the article under investigation; the inert solvent is such that the component is substantially not dissolved, dispersed, disintegrated or denatured by the solvent.

The cylinder is air sealed and left to rest for 1 minute so that the solvent penetrates the whole component. The change in volume is measured and taken as the original volume V_i of the foam specimen. The component is then removed from the solvent and left to dry in air so that the solvent evaporates.

The component is then placed in a 250 ml beaker containing 100 ml of demineralised water, maintained at 25°C, under stirring at 200 rpm with the help of a magnetic stirrer, for 5 minutes. The remaining of the component specimen, if any, is filtered off with a 60mm mesh copper filter and placed in an oven at a temperature and for a period such that residual water is removed. The dried remaining component is re-introduced in the measuring cylinder which volume of acetone had been re-adjusted to 50 ml.

The increase in total volume is monitored and taken as the final volume of the component V_f.. The decrease in total volume ΔV of the component specimen is then:

$$\%\Delta V = \frac{Vf}{Vi} * 100$$

The component preferably has a relative density ρ^* of from 0.01 to 0.95, more preferably from 0.05 to 0.9 or even from 0.1 to 0.8 or even form 0.3 to 0.7. The relative density is the ratio of the density of the component (ρ^*), to the sum of the partial densities of all the bulk materials used to form component(ρ_s).

The preferred foamed component as used herein is air-stable or stable upon contact with air, which means herein that the bulk volume of the component or matrix thereof substantially remains the same when exposed to air. This means in particular that the component retains preferably from 75% to 125% or even from 90% to 110% or even from 95% to 100% of its bulk volume when stored in an open beaker (9 cm diameter; without any protective barrier) in a incubator under controlled ambient conditions (humidity = RH 60%, temperature = 25°C) for 24 hours. Preferably the component retains from 75% to 125% or even from 90% to 110% or even from 95% to 100% of its bulk volume under the above storage conditions whereby the humidity is 80%.

The bulk volume change can be measured by any conventional method. In particular useful is a digital image recorder system containing a digital camera coupled to a personal computer itself equipped with calibrated image analyser software. A $1cm^3$ specimen of the component is obtained and introduced in an open beaker having a diameter of 9 cm and stored for 24 hours at the above conditions. After 24 hours, the size in all three dimensions is measured with the image analysis recorder system. Each specimen measurement is repeated three times, and the average bulk volume change is calculated in %.

Preferably, the component is such that, when in the form of particles of a mean particle size of 2000 microns or less, these particles also retain from 75% to 125% or even from

90% to 110% or even from 95% to 100% of their bulk volume. This can for example be measured by placing 20 grams of such particles, or a weight comprising more than 500 particles, in a volumetric beaker having a diameter of 9 cm. The beaker is taped lightly on its base until the particles re-arrange themselves in a stable position with a horizontal top surface. The volume is measured. The open beaker with the particles is then carefully placed in the incubator for 24 hours, set to the desired %RH and temperature. The bulk volume after the 24 hours is measured and the change of bulk volume is calculated in %.

The component comprises (by weight) preferably at least 1% active ingredient(s), more preferably from 5% to 70%, more preferably at least 10% by weight of the component, more preferably from 15% or even 20% or even 25% to 50%.

The component comprises (by weight) preferably from 10% to 99% matrix, more preferably at least 20% or even 30% to 99%, more preferably from 20% or 30% to 90% to 80%.

The component comprises (by weight) at least 1% dissolution aid, more preferably from 5%, or from 10%, or from 15%, or from 20%, and to 50%, or to 40%, or to 30%, or to 25%.

Matrix

The matrix of the component of the present invention, herein referred to as "matrix", is formed form a polymeric material and a plasticiser. Said polymeric material and said plasticiser are described in more detail hereinafter.

The ratio of plasticiser to polymeric material in the matrix is preferably 1 to 100, more preferably 1 to 70 or 1 to 50, more preferably 1 to 30 or even 1 to 20, depending on the type of plasticiser and polymeric material used. For example, when the polymeric material comprises PVA and the plasticiser comprises glycerine or glycerol derivatives and optionally water, the ratio is preferably around 1:15 to 1:8, a preferred ratio being

around 10:1.

The matrix herein may further comprise the active ingredient of the component herein and/or the dissolution aid of the component herein. Said active ingredient and said dissolution aid are described in more detail hereinafter. Cross-linking agents may also be added to modify the properties of the matrix or the resulting component as appropriate. Borate may be useful in the matrix herein.

The matrix herein preferably has a glass transition temperature (Tg) of below 50°C, preferably below 40°C, preferably less than 20°C or even less than 10°C or even less than 0°C. Preferably the matrix herein has a Tg of above –20°C or even above –10°C.

The Tg of the matrix when used herein, is the Tg of the matrix as present in the component, which thus may be a mixture of polymeric material and plasticiser alone, or a mixture of polymeric material, plasticiser, active ingredient and/or dissolution aid, and in any case, optional additional ingredients may be present (such as, stability agents, densification aids, fillers, lubricants etc., as described hereinafter).

The Tg as used herein is as defined in the text book 'Dynamic Mechanical Analysis' (page 53, figure 3.11c on page 57), as being the temperature of a material (matrix) where the material (matrix) changes from glassy to rubbery, namely where chains gain enough mobility to slide by each other.

The Tg of the matrix of the component of the invention can be measured in the Perkin-Elmer DMA 7e equipment, following the directions in operations manual for this equipment, generating a curve as illustrated in the book Dynamic Mechanical Analysis - page 57, figure 3-11c. The Tg is the temperature or log Frequency as measured with this equipment, between the glass and 'leathery region', as defined in that text.

The matrix, and preferably the component as a whole, has a specific elasticity and flexibility, because of its specific glass transition temperature. In particular, this means that the matrix and the component reversibly deform, absorbing the energy of impacts or

of forces so that the component or matrix remains substantially its original bulk volume after the physical force seizes to be applied on the component.

The elasticity can be defined by the elastic modulus of the matrix, or even the component, which again can be defined by the Young's modulus. This can be calculated from strain or stress mechanical tests as known in the art, for example by using Perkin-Elmer DMA 7e equipment following the manufacturer's experimental procedure over a specific % static strain range, namely in the range of 10-40% static strain. This represents a maximum strain as could be applicable during normal manufacturing or handling. Thus, the elastic modulus as defined herein is the maximum modulus as measured with this equipment in the range of 10% to 40% static strain. For example a piece of matrix (or component) of 1 cm^3 can be used in the testing with this equipment.

The matrix herein typically has an elastic modulus or Young's modulus of less than 4 $GN.m^{-2}$, or typically less than 2 $GN.m^{-2}$, even more preferentially less than 1 $GN.m^{-2}$, but typically even less than 0.5 GN.m¯2, or even less than 0.1 GN.m¯2, or even less than 0.01 $GN.m^{-2}$, as measured with the Perkin-Elmer DMA 7e equipment. In particular a matrix herein which contains gas bubbles, e.g. formed by processes involving the introduction of air in the matrix, has an elastic modulus below 0.1 $GN.m^{-2}$ or even 0.01 $GN.m^{-2}$ or even below 0.005 $GN.m^{-2}$ or even below 0.0001 $GN.m^{-2}$.

Preferably the matrix is flexible, such that it has a relative yield strain greater than 2%, and preferably greater than 15% or even greater than 50%, as measured with the Perkin-Elmer DMA 7e equipment. (The yield strain is in this measurement the limit deformation of a piece of matrix at which the it deforms irreversible).

In particular this means that when a matrix sample having a cross section of a specific length, for example 1cm, is compressed with a static force applied along the axis of that cross section, the static force being variable but at least equivalent to twice atmospheric pressure, the change of this length after removal of the force is at least 90% to 110% of

the original length. This can for example be measured by use of Perkin-Elmer DMA 7e equipment.

Similarly, the matrix is preferably flexible to such an extend that when a matrix sample having a cross section of a specific length, for example 1cm, is stretched with a static force applied along the axis of that cross section, the static force being variable, but at least equivalent to twice atmospheric pressure, the change of this length after removal of the force is at least 90% to 110% of the original length. This can for example be measured by use of Perkin-Elmer DMA 7e equipment.

In particular, when using this equipment, the static forces applied along the axis of a cross section of a 1 cm^3 matrix sample are gradually increased until the deformation of the component, in the direction of the cross section, is 70%. Then, the force is removed and the final deformation of the matrix sample in the direction of the cross section is measured. Preferably, this length of the cross section after this experiment is preferably from 90% to 110% of the original length of the cross section, preferably from 95% to 105% or even from 98% to 100%.

The elastic modulus or Young's modulus is related to the relative density, namely

$$\frac{E^*}{E_S} \approx \left(\frac{\rho^*}{\rho_S}\right)^2,$$

where ρ^* is the relative density of the matrix or even the component, and ρ_s is the relative densities of the components of the matrix or component, as described herein, and E^* is the Young's modulus of the matrix or even the component itself, and E_s that of the components of the matrix or even the component. This means that even a stiff polymeric material, with a high E_s can be made into an elastic, flexible matrix by adjusting the levels and/ or type of plasticiser and optionally by modifying the density (or for example by introducing gas during the making process to form foam component, as described below).

The matrix, or even the component as a whole, is in the form of a foam and preferably such that it forms an interconnected network of open and/ or closed cells, in particular a network of solid struts or plates which form the edges and faces of open and/ or closed cells. The spacing inside the cells can contain part of the active ingredient and/ or a gas, such as air.

Preferably, the ratio of the closed cells to open cells in the matrix of the component, or the component as a whole is more than 1:1, preferably more than 3:2 or even more than 2:1 or even more than 3:1. This ratio can be determined by calculating the Total Volume of a specimen of the matrix or component, V_T , (assuming a spherical shape), and then measuring with a Mercury Porosimetry Test method the Open Cell Volume (V_O) and subtracting the Open Cell Volume from the Total Volume should deliver the Closed Cell Volume (V_C: $V_T = V_O + V_C$).

<u>Polymeric material</u>

Any polymeric material can be used to form the matrix herein, preferably the polymeric material has itself a Tg as described above or more typically, it can be formed into a matrix having the Tg as described above by using a suitable amount of plasticiser.

Preferably, the polymer material comprises or consists of amorphous polymer(s).

The polymeric material may consist of a single type of homologous polymer or may be a mixture of polymers. Mixtures of polymers may in particular be beneficial to control the mechanical and/or dissolution properties of the component, depending on the application thereof and the requirements thereof.

Preferred it that the polymeric material comprises a water-dispersible or more preferably a water-soluble polymer. Water-dispersible and water-soluble are typically defined as described hereinbefore, as per the method for determining the water-solubility and water-dispersibility of the component herein. Preferred water-dispersible polymers herein have

a dispersibility of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out hereinbefore using a glass-filter with a maximum pore size of 50 microns; more preferably the polymer herein is a water-soluble polymer which has a solubility of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out hereinbefore using a glass-filter with a maximum pore size of 20 microns.

The polymer can have any average molecular weight, preferably from about 1000 to 1,000,000, or even form 4000 to 250,000 or even form 10,000 to 200,000 or even form 20,000 to 75,000. Highly preferred may be polymeric material having a weight average molecular weight of from 30,000 to 70,000.

Depending on the required properties of the component herein, the polymeric material can be adjusted. For example, to reduce the solubility, polymers may be included in the material, which have high molecular weights typically above 50,000 or even above 100,000, and vice versa. For example, to change the solubility, polymers of varying level of hydrolyses may be used. For example, to improve (reduce) the elastic modulus, the cross-linking of the polymers may be increased and/ or the molecular weight may be increased.

It may be preferred that the polymer used in the component herein has a secondary function, for example a function in the composition wherein component is to be incorporated. Thus, for example, for cleaning products, it is useful when the polymer in the polymeric material is a dye transfer inhibiting polymer, dispersant etc.

Preferred are polymers selected from polyvinyl alcohols and derivatives thereof, polyethylene glycols and derivatives thereof, polyvinyl pyrrolidone and derivatives thereof, cellulose ethers and derivatives thereof, and copolymers of these polymers with one another or with other monomers or oligomers. Most preferred are PVP (and derivatives thereof) and/ or PEG (and derivatives thereof) and most preferably PVA (and

derivatives thereof) or mixtures of PVA with PEG and/ or PVP (or derivatives thereof). Most preferred may also be a polymeric material only comprising PVA.
Preferably, such polymers have a level of hydrolysis of at least 50%, more preferably at least 70% or even from 85% to 95%.

Plasticiser

Any plasticiser which is suitable to aid the formation of a matrix as defined herein can be used. Mixtures of plasticiser may also be used.

Preferably, the plasticiser or at least one of the plasticisers, has a boiling point above 40°C, preferably above 60°C, or even above 95°C, or even above 120°C, or even above 150°C.

Preferred plasticisers include glycerol or glycerine, glycol derivatives including ethylene glycol, digomeric polyethylene glycols such as diethylene glycol, triethylene glycol and tetraethylene glycol, polyethylene glycol with a weight average M.W. of below 1000, wax and carbowax, ethanolacetamide, ethanolformamide, triethanolamine or acetate thereof, and ethanolamine salts, sodium thiocyanates, ammonium thiocyanates, polyols such as 1,3-butanediol, sugars, sugar alcohols, ureas, dibutyl or dimethyl pthalate, oxa monoacids, oxa diacids, diglycolic acids and other linear carboxylic acids with at least one ether group distributed along the chain thereof, water or mixtures thereof.

Preferably, when water is used, an additional plasticiser is present. If water is used, then the water is typically present in the foam component at a level of at least 3 wt%, preferably more than 3 wt%, preferably even at least 5 wt%, or even at least 10 wt%.

The plasticiser is preferably present at a level of at least 0.5% by weight of the article, preferably by weight of the matrix, provided that when water is the only plasticiser it is present at a level of at least 3% by weight of the component, or preferably by weight of the matrix.

Preferably, the plasticiser is present at a level of 1% to 35% by weight of the article or matrix, more preferably 2% to 25% or even to 15% or even to 10% or even to 8% by weight of the article or by weight of the matrix. The exact level will depend on the polymeric material and plasticiser used, but should be such that the matrix of the article has the desired Tg. For example, when urea is used, the level is preferably 1% to 10% by weight of the matrix, while when glycerine or ethylene glycol or other glycol derivatives are used, higher levels may be preferred, for example 2% to 15% by weight of the component or matrix.

Active ingredient

The active ingredient can be any material which is to be delivered to a liquid environment, or preferably an aqueous environment and preferably an ingredient which is active in an aqueous environment. For example, when used in cleaning compositions the component can contain any active cleaning ingredients. The component may also comprise compositions, such as cleaning composition or personal care compositions.

In particular, it is beneficial to incorporate in the component, active ingredients which are moisture sensitive or react upon contact with moisture, or solid ingredients which have a limited impact robustness and tend to form dust during handling.

The active ingredient is typically a moisture sensitive ingredient, a temperature sensitive ingredient, an oxidizeable ingredient, a volatile ingredient, or a combination thereof. The active ingredient may be biological viable material, hazardous or toxic material an agricultural ingredient such as an agrochemical, a pharmaceutical ingredient such as a medicine or drug, or a cleaning ingredient.

In particular preferred in component are active ingredients, such as enzymes, perfumes, bleaches, bleach activators, fabric cationic and/or silicone softeners and/or conditioners, antibacterial agents, brighteners, photo-bleaches and mixtures thereof.

Another active ingredient is a perhydrate bleach, such as metal perborates, metal percarbonates, particularly the sodium salts. Also preferred active ingredients are organic peroxyacid bleach precursor or activator compound, preferred are alkyl percarboxylic precursor compounds of the imide type include the N-,N,N^1N^1 tetra acetylated alkylene diamines wherein the alkylene group contains from 1 to 6 carbon atoms, particularly those compounds in which the alkylene group contains 1, 2 and 6 carbon atoms such as tetraacetyl ethylene diamine (TAED), sodium 3,5,5-tri-methyl hexanoyloxybenzene sulfonate (iso-NOBS), sodium nonanoyloxybenzene sulfonate (NOBS), sodium acetoxybenzene sulfonate (ABS) and pentaacetyl glucose, but also amide substituted alkyl peroxyacid precursor compounds

Highly preferred active ingredient for use in the component herein are one or more enzymes. Preferred enzymes include the commercially available lipases, cutinases, amylases, neutral and alkaline proteases, cellulases, endolases, esterases, pectinases, lactases and peroxidases conventionally incorporated into detergent compositions. Suitable enzymes are discussed in US Patents 3,519,570 and 3,533,139. Preferred commercially available protease enzymes include those sold under the tradenames Alcalase, Savinase, Primase, Durazym, and Esperase by Novo Industries A/S (Denmark), those sold under the tradename Maxatase, Maxacal and Maxapem by Gist-Brocades, those sold by Genencor International, and those sold under the tradename Opticlean and Optimase by Solvay Enzymes. Preferred amylases include, for example, α-amylases obtained from a special strain of B licheniformis, described in more detail in GB-1,269,839 (Novo). Preferred commercially available amylases include for example, those sold under the tradename Rapidase by Gist-Brocades, and those sold under the tradename Termamyl, Duramyl and BAN by Novo Industries A/S. Highly preferred amylase enzymes maybe those described in PCT/ US 9703635, and in WO95/26397 and WO96/23873. The lipase may be fungal or bacterial in origin being obtained, for example, from a lipase producing strain of Humicola sp., Thermomyces sp. or Pseudomonas sp. including Pseudomonas pseudoalcaligenes or Pseudomas fluorescens. Lipase from chemically or genetically modified mutants of these strains are also useful

herein. A preferred lipase is derived from <u>Pseudomonas</u> <u>pseudoalcaligenes</u>, which is described in Granted European Patent, EP-B-0218272.
Another preferred lipase herein is obtained by cloning the gene from <u>Humicola</u> <u>lanuginosa</u> and expressing the gene in <u>Aspergillus</u> <u>oryza</u>, as host, as described in European Patent Application, EP-A-0258 068, which is commercially available from Novo Industri A/S, Bagsvaerd, Denmark, under the trade name Lipolase. This lipase is also described in U.S. Patent 4,810,414, Huge-Jensen et al, issued March 7, 1989.

<u>Dissolution aid</u>

The component of the invention comprises a dissolution aid.

The dissolution aid is in addition to the active ingredient of the component herein. If more than one active ingredient is comprised by the component herein, then it may be preferred that one of the active ingredients is selected such that is, or acts as, a dissolution aid. For the purpose of the present invention, the dissolution aid is always an additional ingredient of the component herein to the active ingredient of the component herein.

Therefore, for the purpose of the present invention, for a component to be defined as comprising a dissolution aid and an active ingredient, wherein said component comprises an ingredient having dual functionality and can function as an active ingredient or a dissolution aid as defined herein, an additional dissolution aid or active ingredient must be present in the article herein, which is in addition to the ingredient having dual functionality, to obtain a component comprising an active ingredient and a dissolution aid which is in accord with the present invention.

The above statements with regard to ingredients having dual functionality are also true when considering other embodiments of the present invention. It is essential for the component herein to comprise an active ingredient, a polymeric material and a plasticiser (which form the matrix), and a dissolution aid. Thus, to obtain a component in accord with the present invention, at least four different ingredients must be present in said

component. It may be preferred to select essential ingredients which have dual functionality.

The above statements with regard to dual functionality ingredients are also true when considering highly preferred embodiments of the present invention, for example preferred components comprising additional ingredients such as a stabilising aid.

The dissolution aid may preferably comprise a sulfonated compound such as C_1-C_4 alk(en)yl sulfonates, C_1-C_4 aryl sulfonates, di iso butyl benzene sulphonate, toluene sulfonate, cumene sulfonate, xylene sulfonate, salts thereof such as sodium salts thereof, derivatives thereof, or combinations thereof, preferably di iso butyl benzene sulphonate, sodium toluene sulfonate, sodium cumene sulfonate, sodium xylene sulfonate, and combinations thereof.

The dissolution aid may comprise a C_1-C_4 alcohol such as methanol, ethanol, propanol such as iso-propanol, and derivatives thereof, and combinations thereof, preferably ethanol and/or iso-propanol.

The dissolution aid may comprise a C_4-C_{10} diol such as hexanediol and/or cyclohexanediol, preferably 1,6-hexanediol and/or 1,4-cyclohexanedimethanol.

The dissolution aid may comprise compounds which are capable of acting as whicking agents, such as cellulosic based compounds, especially modified cellulose.

The dissolution aid may comprise swelling agents such as clays. Preferred clays are smectite clays, especially dioctahedral or trioctrahedral smectite clays. Highly preferred clays are montmorillonite clay and hectorite clay, or other clays found in bentonite clay formations.

The dissolution aid preferably comprises an effervescence system. A preferred effervescence system comprises an acid source capable of reacting with an alkali source

in the presence of water to produce a gas. The gas produced by this interaction, includes nitrogen, oxygen and carbon dioxide gas. The acid source may be any organic, mineral or inorganic acid, or a derivative thereof, or a mixture thereof. Preferably the acid source comprises an organic acid. Suitable acid sources include citric, malic, maleic, fumaric, aspartic, glutaric, tartaric succinic or adipic acid, monosodium phosphate, boric acid, or derivatives thereof. Citric acid, maleic or malic acid are especially preferred.

As discussed hereinbefore, the effervescence system preferably comprises an alkali source, however, for the purpose of the invention, it should be understood that the alkali source may be part of the component or can be part of a composition comprising the component, or can be present in the washing liquor, whereto the component, or a composition comprising the component, is added. Any alkali source which has the capacity to react with the acid source to produce a gas may used herein. Preferred alkali sources can be perhydrate bleaches, including perborate, and silicate material.

Preferably the gas is carbon dioxide, and therefore the alkali source is a preferably a source of carbonate, which can be any source of carbonate known in the art. In a preferred embodiment, the carbonate source is a carbonate salt. Examples of preferred carbonates are the alkaline earth and alkali metal carbonates, including sodium or potassium carbonate, bicarbonate and sesqui-carbonate and any mixtures thereof with ultra-fine calcium carbonate such as are disclosed in German Patent Application No. 2,321,001 published on November 15, 1973. Alkali metal percarbonate salts are also suitable sources of carbonate species, which may be present combined with one or more other carbonate sources.

The molecular ratio of the acid source to the alkali source present in the component is preferably from 50:1 to 1:50, more preferably from 20:1 to 1:20 more preferably from 10:1 to 1:10, more preferably from 5:1 to 1:3, more preferably from 3:1 to 1:2, more preferably from 2:1 to 1:2.

<u>Additional Ingredients</u>

The component of the invention preferably comprises additional ingredients which can improve the stability of the active ingredient of the article herein.

These additional ingredients are typically capable of stabilising the active ingredient of the component herein, this is especially preferred when the active ingredient(s) comprise an oxidative or moisture sensitive active ingredient, such as one or more enzymes. These additional ingredients may also stabilise the matrix of the component herein, and thus indirectly stabilise the active ingredient. These stabilising ingredients are defined herein as “stabilising agents”.

The stabilising agent is preferably a compound which stabilises the active ingredient, or matrix, from oxidative and/or moisture degradation during storage. The stabilising agent may be, or comprise, a foam matrix stabiliser. The stabilising agent may be, or comprise, an active ingredient stabiliser, especially an enzyme stabiliser. Stabilising agents which are capable of stabilising the active ingredient indirectly by keeping the foam matrix of the article stable, herein referred to as “foam stabiliser”.

Foam stabilisers preferably comprise a surfactant such as a fatty alcohol, fatty acid, alkanolamide, amine oxide, or derivatives thereof, or combinations thereof. The foam stabiliser may comprise betaine, sulfobetaine, phosphine oxide, alkyl sulfoxide, derivatives thereof, or combinations thereof.

Other preferred foam stabilisers comprises one or more anions or cations such as mono-, di-, tri- valent, or other multivalent metal ions, preferred are salts of sodium, calcium, magnesium, potassium, aluminium, zinc, copper, nickel, cobalt, iron, manganese and silver, preferably having an anionic counterion which is a sulphate, carbonate, oxide, chloride, bromide, iodide, phosphate, borate, acetate, citrate, and nitrate, and combinations thereof.

The foam stabiliser may comprise finely divided particles, preferably finely divided particles having an average particle size of less than 10 micrometers, more preferably less than 1 micrometer, even more preferably less than 0.5 micrometers, or less than 0.1 micrometers. Preferred finely divided particles are aluminosilicates such as zeolite, silica, or electrolytes described hereinbefore being in the form of finely divided particles.

The foam stabiliser may comprise agar-agar, sodium alginate, sodium dodecyl sulfate, polyethylene oxide, guar gum, polyacrylate, or derivatives thereof, or combinations thereof.

The foam stabiliser may be coating which is separate to the matrix of the article herein. The foam stabiliser typically partially encloses, preferably completely encloses, the article herein or the active ingredient thereof.

The coating is typically contacted to, preferable in such a manner as to form a coat on, the active ingredient prior to said active ingredient being contacted to the polymeric material or the plasticiser of the matrix, and preferably being incorporated in the article herein.

The coating may typically be contacted to, preferable in such a manner as to form a coat on, the article herein subsequent to the polymeric material and the plasticiser forming the matrix, and preferably subsequent to the active ingredient contacting said matrix or being incorporated in the article herein.

Preferred coating comprises polymers, typically selected from polyvinyl alcohols and derivatives thereof, polyethylene glycols and derivatives thereof, polyvinyl pyrrolidone and derivatives thereof, cellulose ethers and derivatives thereof, and copolymers of these polymers with one another or with other monomers or oligomers. Most preferred are PVP (and derivatives thereof) and/ or PEG (and derivatives thereof) and most preferably PVA (and derivatives thereof) or mixtures of PVA with PEG and/ or PVP (or derivatives thereof). These polymers do not form the matrix of the article herein. Thus, these polymers are different to the polymeric materials of the foam matrix.

A preferred coating comprises compounds such as glycerol or glycerine, glycol derivatives including ethylene glycol, digomeric polyethylene glycols such as diethylene glycol, triethylene glycol and tetraethylene glycol, polyethylene glycol with a weight average M.W. of below 1000, wax and carbowax, ethanolacetamide, ethanolformamide, triethanolamine or acetate thereof, and ethanolamine salts, sodium thiocyanates, ammonium thiocyanates, polyols such as 1,3-butanediol, sugars, sugar alcohols, ureas, dibutyl or dimethyl pthalate, oxa monoacids, oxa diacids, diglycolic acids and other linear carboxylic acids with at least one ether group distributed along the chain thereof, water or mixtures thereof. These compounds do not form the foam matrix of the article herein. Thus, these compounds are different to the plastisicer of the foam matrix.

Preferred stabilising agents that are capable of stabilising the active ingredient directly, especially if said active ingredient comprises one or more enzymes, are defined herein as "active stabilisers" or "enzyme stabilisers". Typically active stabilisers interact directly with, and stabilise, the active ingredient.

Typical active stabilisers for use herein preferably comprise a surfactant. Suitable surfactants for use herein are those described hereinbefore as surfactants suitable for use as matrix stabilisers. In addition to these surfactants, other surfactants suitable for use herein may comprise surfactants such as sodium alky(en)yl sulfonates, sodium alkoxysulfonates, preferred alkoxysulfonates are those comprising from 10 to 18 carbon atoms in any conformation, preferably linear, and having an average ethoxylation degree of from 1 to 7, preferably from 2 to 5.

Other preferred active stabilisers comprise boric acid, formic acid, acetic acid, and salts thereof. These acid salts preferably comprise counerions such as calcium and/or sodium.

Preferred active stabilisers comprise cations such as calcium and or sodium. Preferably calcium chloride and/or sodium chloride.

Other preferred active stabilisers comprise small peptide chains averaging from 3 to 20, preferably from 3 to 10 amino acids, which interact with and stabilise the active ingredient, especially enzyme(s).

Other active stabilisers comprise small nucleic acid molecules, typically comprising from 3 to 300, preferably from 10 to 100 nucleotides. Typically nucleic acid molecules are deoxyribonucleic acid and ribonucleic acid. The nucleic acid molecules may be in the form of a complex with other molecules such as proteins, or may form a complex with the active ingredient of the article herein, especially enzyme(s).

Active stabilisers suitable for use herein, especially when the article herein comprises a bleach, comprise anti-oxidants and/or reducing agents such as thiosulphate, methionine, urea, thiourea dioxide, guanidine hydrochloride, guanidine carbonate, guanidine sulfamate, monoethanolamine, diethanolamine, triethanolamine, amino acids such as glycine, sodium glutamate, proteins such as bovine serum albumin and casein, tert-butylhydroxytoluene, 4-4,-butylidenebis (6-tert-butyl-3-methyl-phenol), 2,2'-butlidenebis (6-tert-butyl-4-methylphenol), (monostyrenated cresol, distyrenated cresol, monostyrenated phenol, distyrenated phenol, 1,1-bis (4-hydroxy-phenyl) cyclohexane, or derivatives thereof, or a combination thereof.

Other active stabilisers may comprise a reversible inhibitor of the active ingredient. Without wishing to be bound by theory, it is believe that a reversible inhibitor of the active ingredient, especially if the active ingredient comprises one or more enzymes, may form a complex with, and improve the stability of, said active ingredient, and thus, stabilises the active ingredient during storage. When the active ingredient is released, typically into a liquid environment, the reversible inhibitor dissociates from the active ingredient and the active ingredient is then able to perform the desired action it is designed or intended to perform.

Active stabilisers suitable for use herein may comprise sugars, Typical sugars for use herein include those selected from the group consisting of sucrose, glucose, fructose, raffinose, trehalose, lactose, maltose, derivatives thereof, and combinations thereof.

The active stabiliser may also comprise sugar alcohols such as sorbitol, mannitol, inositol, derivatives thereof, and combinations thereof.

It may be preferred that the active stabiliser is in the form of a coating or barrier which at least partially encloses the article herein or the active ingredient thereof, preferably completely encloses the article herein or the active ingredient thereof, especially an enzyme.

Process for making foam component

The component of the invention can be made by any process of making a polymer matrix of the defined Tg from a polymeric material and a plasticiser, and combining an active ingredient and a stabilising agent with such a matrix. Preferred processes involve chemically or physically introducing a gas in a mixture of the polymeric material and plasticiser and optionally the active ingredient.

A preferred process for making the component herein comprising the step of

a) obtaining a mixture of a polymeric material and a plasticiser, preferably water and an additional plasticiser;
b) chemically or physically introducing gas in said mixture of polymeric material and water;
c) prior to step b) and/or simultaneously with step b) and/ or subsequently to step b), addition of the active ingredient to said mixture;
d) prior to step c) and/or simultaneously with step c) and/ or subsequently to step c), contacting a dissolution aid to said mixture;
e) shaping the articles of the resulting mixture;

whereby one or more of steps a) to e) are followed or accompanied by removal of part of the water, if present.

In step a) the mixture is preferably an aqueous mixture or slurry and after or in step b), c) and/ or d), part of the water is removed such that the resulting component comprises 3% by weight of free-moisture, or more.

Step c) preferably comprises the step of obtaining a body comprising the active ingredient or part thereof and enclosing said body with the mixture of step b).

Step d) preferably comprises the step of mixing, more preferably intimately mixing or enclosing, the active ingredient with the stabilising agent prior to contacting said stabilising agent, and preferably said active ingredient, to said mixture.

Preferably, the component comprises open and/ or closed cells and the process comprising the steps of

a) formation of a mixture of the polymeric material, the active material, a dissolution aid, a plasticiser and a liquid, whereby the liquid and the plasticiser may be the same compound;
b) shaping of bodies from the mixture of claim b) and
c) evaporation of the liquid or part thereof to form spacings in the mixture which form the inner area of the cells of the component,

whereby step c) is preferably conducted by freeze drying or by heating the bodies, thereby causing the liquid or part thereof to evaporate.

Step b) may also be conducted by submitting the mixture of a) to pressure, preferably under mixing and/ or increasing the temperature, and subsequently removing the pressure or part thereof, thereby causing the liquid to evaporate. For example, an extrusion process can be used. Hereby it is preferred that the mixture of the polymeric material, plasticiser, preferably including water, and optionally the active ingredient and/or dissolution aid, is introduced in an extruder, wherein the mixture is further mixed and heated, due to the

mixing or due to applying heat, preferably such that the mixture therein forms a melt, and then dropping the pressure at the exit point where the extruded mixture (which can be formed into the desired form, for example granules) exits the extruder, whereby the liquid or part thereof evaporates, or preferably the water evaporates as steam from the extruded mixture. This results in formation of cells with spacings, as described above, which then may contain a gas, preferably air, and optionally the active ingredient. These spacings form the internal area of the cells of the matrix of the component of the invention.

Step b) in the process may also be conducted by heating the mixture to cause the liquid or part thereof to evaporate, resulting in the formation of spacings, as above. This can preferably done by feeding the mixture into a spray drying tower, preferably such that the mixture is fed through spray nozzles which form droplets of the mixture, and spray drying the droplets at conventional temperature, resulting in the component.

The physical and/ or chemical introduction of gas or foaming, as mentioned above can be done by any known method, preferred are

- physical foaming by gas injection (dry or aqueous route) optionally under mixing, high shear stirring (dry or aqueous route), gas dissolution and relaxation including critical gas diffusion (dry or aqueous route);
- chemical foaming by in-situ gas formation (via chemical reaction of one or more ingredients, including formation of CO_2 by an effervescence system),
- steam blowing, UV light radiation curing.

These foaming steps are preferably followed by a drying step or an additional drying step to remove excess liquid or part thereof, such as water. In particular, the drying step is at least done after the polymer matrix is formed, and optionally after the active ingredient is added, preferably as final step in the process. The drying step is done preferably such that the final component is of about the same volume after the drying step as before the drying step. Thereto, the drying step is preferably done by freeze-drying, whereby the solvent, e.g. water, is removed under vacuum and reduced temperatures. Also useful can

be slow fluid bed drying or oven drying at modestly increased temperatures, such as 40-80°C, or even 40-60°C .

Preferred processes involve at least the step of formation of a mixture of polymeric material and a liquid, preferably a solution of polymeric material and a solvent, preferably comprising water, and adding a plasticiser (or as the case may be, additional plasticiser) to this. If the presence of the active ingredient and/or the dissolution aid in the matrix is required, these are also added to the mixture of polymeric material solvent and plasticiser. Alternatively, or in addition, it may be preferred that the matrix is formed around the active material, preferably a core of the active material and carrier material.

Preferred means of incorporating the dissolution aid into the component herein include;
a) contacting, preferably incorporating therein, a dissolution aid to a dry foam component to obtain a foam component in accord with the present invention, e.g. this can be for example by injecting, or enclosing the matrix of the component; and/or
b) cooling the foam component to a temperature near, preferably just above, to said foam components freezing point, and subsequently contacting or incorporating the dissolution aid to said foam component to obtain a foam component in accord with the present invention; and/or
c) as b), followed by freezing said foam component, for example prior to freeze drying; and/or
d) partially coating, or preferably coating, the dissolution aid with a material such as cetyl alcohol.

This is than further processed for example into bodies of the shape of the final component, for example particles or beads, and typically dried to obtain the components. Preferably, a gas is added prior to the shaping step. Shaping steps include granulation steps such as atomisation or spray-drying, extrusion, micro pastillisation. Freeze drying is a preferred process to dry the bodies to form the components.

The following are preferred processes resulting in low dust or even nil dust particles, as measured with the stressed Heubach test described below, having a matrix with a Tg of below 10°C and an elastic modulus of below $0.5GNm^{-2}$, as indicated in more detail in the following specific examples.

A first preferred process is as follows:

The required amount of a solution of the polymer material (or a mixture of polymer and a liquid) is obtained, and for example (introduced) in a mix tank. Then the required amount of (a solution of) the active material, for example an enzyme solution, and dissolution aid is added, and the required amount of plasticiser is added, and optionally other additional ingredients, such as fillers, densification agents etc. This is agitated to become a homogeneous mixture. Preferably, a gas such as air can be introduced into the solution, by any of the methods above, preferably physically, by high sheer mixing.

Then, particles are formed from this mixture by atomisation, preferably using a Positive Displacement pump to transfer the mixture solution to a spray nozzle (s), preferably using either single or multi-fluid nozzles to create liquid droplets.

The liquid droplets are then frozen, preferably by passing through a refrigeration media (can include liquid nitrogen, freon, refrigeration oils). Then, the frozen particles are transferred to a vacuum chamber, preferably having a temperature (as measured on the surface of the particles) below 0°C.

The frozen particles are preferably collected from the spray column and transferred without raising the temperature. The temperature of the walls and contact trays of the freeze dryer are preferably maintained below 0°C to keep the particles frozen.

A vacuum is applied, and the frozen ice crystals will sublimate a gas form, resulting in cells in the particle. The total drying degree can be controlled with the level of vacuum, and contact temperature of the chamber walls and trays.

After the particles have been dried to the desired moisture content, they will be free flowing. Then preferably, the particles can be classified via a variety of screens and or process equipment.

The optional step above, of introducing gas (bubbles) into the polymeric solution mixture

has been found to give a much better impact resistance to the particle, reflected by its electric modules. The introduction of gas bubbles can be accomplished in a variety of ways.

In the atomisation step, the atomizing nozzle should preferably be located in a spray column with sufficient height to accomplish droplet freezing while gravity falling. The nozzle type can be of various designs - single fluid pressure nozzle, spinning insert, sonic, or multi-fluid nozzle. The important aspect is to disrupt the liquid stream to form discrete liquid droplets. As these droplets fall with gravity, they need to be cooled to freezing. The freezing media is preferably non-aqueous gas or liquid which can provide rapid freezing of the liquid droplets. The actual temperatures for cooling these droplets to form particles, is preferably below 0°C and preferably below -20°C.

It may also be preferred that the above process is modified as follows: a gas, preferably CO_2 gas, is introduced in the mixture and the mixture is introduced in a spray drying tower, as above, thereby forming spray-dried foamed particles, which can be classified if necessary. Preferably the inlet temperature in the tower is about 130°C and the outlet temperature about 75°C and the spray rate is 12.5g/min. For example a Niro Mobil Minor with two fluid nozzles can be used hereby. The resulting particle may be already of the required form, or may be submitted to further freeze drying under vacuum.

<u>Another preferred process is as follows:</u>

The required amount of a solution of the polymer material is obtained (or alternatively, a powdered polymer can be used provided a liquid is added) and for example introduced in a mix tank. Then the required amount of plasticiser is added, and optionally other additional ingredients, such as fillers, densification agents etc. This is agitated to become a homogeneous mixture. Preferably, a gas such as air can be introduced into the solution, by any of the methods above, preferably physically, by high sheer mixing.

Also, particles comprising the active ingredient, such as an enzyme, and the dissolution aid, and optionally other ingredients, such as fillers or carriers are prepared, for example

by fluid bed coating, by charging first 'cores' (typically when the actives care enzymes, these core particles are sugar or starch particles), to a fluid bed and spraying the active material or a solution of the active material onto these cores and then drying any solvent such as water from the active solution off with warm fluidizing air.

Then, the polymeric mixture above is introduced onto these active/cores , for example via a positive displacement pump leading to an atomizing nozzle inside the fluid bed as described above. More than one nozzle can be used and it may be preferred that different ingredients are added to the core via different nozzles.

The fluidizing air needs is preferably below 0°C, preferably around -20°C. Then, the fluidizing air freezes the polymeric mixture/solution onto the outside of the active-core. This is a critical parameter to control and typically the air temperature must be below 0°C in order to quickly freeze the polymeric mixture/solution onto the core particles.

Then preferably, the frozen particles thus obtained are transferred to a vacuum chamber, as above, and also classification may take place.

This technology results in particle of the invention comprising a matrix around the active ingredient.
The optional step above, of introducing gas (bubbles) into the polymeric solution mixture has been found to give a much better impact resistance to the particle, reflected by its Young's Modulus.

Another preferred process is as follows:

The required amount of a solution of the polymer material (or solid polymer and a suitable amount of liquid) is obtained, and for example introduced in a mix tank. Then the required amount of (a solution of) the active material, for example an enzyme solution, and a dissolution aid (a solution of) is added (into the mix tank) and the required amount of plasticiser is added, and optionally other additional ingredients, such as fillers, densification agents. This is agitated to become a homogeneous mixture. Preferably, a gas

such as air can be introduced into the solution, by any of the methods above, preferably physically, by high sheer mixing.

Then, the polymeric solution is pumped from the mix tank into an extruder or into a cavity with a die plate at the end. Before entering the extruder or cavity, gas can be injected into the mixture and for example be dispersed via a mechanical shear mixer or a static mixer.
As the extrudate exits the die plate, the change in pressure creates a slight puffing or swelling in the extrudate. The extrudate is then cut to the correct length with either a die face cutter or with some other device (e.g. heated wire, rotating plug cutter, etc.). The extrudates can optionally go through additional rounding steps to become more spherical. Process equipment that can accomplish this function include (rotating pans, agglomeration pans, marumerizers, tumbling drums, mixing drums, etc.).

For example, a paste is prepared by mixing 75g PVOH, 15g Citric acid, 2g PEO and 22.5g glycerol in a Braun mixer, high sheer, namely set at full speed for 40seconds: then 80g H_2O and 80g enzyme was added and mixed at high sheer, namely set at full speed until a smooth foam had formed, approximately within 2mins. The foam was extruded from a 10ml syringe onto a plastic sheet. This was left for 24 hours to dry. Once dry the foam strips were cut into approximately 1 - 2mm sections to form particles of the formula (dry) 63.2 polyvinyl alcohol, 19% glycerol, 12.7|% citric acid, 1.6% PEO, 4% water, 3.2% enzyme.

The resulting particles had an elastic modulus of $0.00016 GN.m^{-2}$.

The resulting particles give 0% dusting when tested in a stressed Heubach test, which indicated a very good impact robustness. (The stressed Heubach test is performed as known in the art, using equipment as supplied by Heubach Engineering GMbH, Germany, with the stressed modification of the rotation speed of the impeller being 75 $\pm$ 1 rpm and the balls being of Tungsten carbide and 82 grams each.).

Preferred moulding / pastillisation process:

A highly preferred process involves shaping particles of mixtures as described above by use of a mould; whereby mixtures as described herein are introduced in a mould and subsequently dried (freeze dried). Also preferred is such a process which uses pastille making equipment, whereby mixtures as described above, preferably comprising also introduced gas, are forced through a rotating perforated drum onto a moving conveyor belt, shaping into pastilles (droplets or particles). When dry or hardened, the thus formed particles or beads are removed from the conveyor belt by a scraper.

Preferably, the first step is to make a mixture of polymeric material and plasticiser, a liquid component, and optionally the active ingredient and dissolution aid. Preferably, gas is introduced into this mixture as described herein. This must preferably be free from large undissolved particles which may block up the perforations in the drum. The mixture is preferably in the temperature range 0-50°C. The mixture is pumped into a manifold that enters the rotating perforated drum and is parallel to the longitudinal axis of the drum. The mixture is pumped into the inner of the drum and, as the drum rotates, is brought into contact with an internal scraper bladed lying in contact and along the length of the inner surface of the perforated drum, parallel to the feed manifold. The distance of the outside surface of the perforated drum is within the height of the desired particle height (which is less than the diameter of the perforations) but not touching a moving conveyor belt or a rotating smooth surfaced drum at the point where the internal scraper is in contact with the inner surface of the perforated drum, the tangential speed of the perforated drum matched by the speed of the conveyor belt or the tangential speed of the smooth surfaced drum. As the mixture is forced through the perforations, which are typically in the size range 300 - 2000μm (but may be smaller or larger), it is deposited onto the surface below. The rotation of the perforated drum shears the feed material away from the material on the smooth surface thus leaving a droplet, or pastille, which will form the required particle. These pastilles can be set by either chilling or by evaporation of some or all of the solvent fluid. If chilling is required the temperature of the conveyor belt or smooth surfaced drum may be in the range ambient to -20°C. If evaporation of a solvent is required, then this can be achieved by heat

conduction from the conveyor belt which may be in the range ambient to 70°C, by drying air (which may be heated up to 200°C to reduce drying time) passing over the surface of the pastilles, or both.

Then, they resulting particles are removed from the drum or conveyor belt by a scraper. This removal process may be improved by the use of a suitable lubricant (release agent) on the drum, such as silicone oil. This lubricant or release agent may show an added benefit to the particle by reducing adhesive properties between the polymeric mixture and the belt/drum and thus increasing the pastille height, if this is a desirable feature.

Examples

Example 1

A process of preparing a foam component in accord with the present invention

4700g of a 33%w/w solution of polyvinyl alcohol (weight average M.W being from 30,000 to 70,000) is mixed with 3360g enzyme solution (5% by weight active enzyme and 85% by weight water), 159.3g of glycerol and 155g of cyclohexane dimethanol in a high shear mixer until a smooth foam is formed. This mixture is transferred into a feed tank, and using a gear pump, it is pumped into micropastillisation equipment, for example as supplied by Sandvik Process Systems, Totowa New York, using a perforated drum with perforations of 1mm diameter, spaced 2.5mm apart. The apparatus deposits pastilles onto a smooth surfaced drum coated with a film of silicone oil and heated to ~30°C. When one quarter of the drum is covered by pastilles, the drum is stopped from rotating. The pastilles are dried using a hot air heater until the surfaces of the pastilles are dry to the touch. The resulting particles are then scraped off and collected.

Example 2

A process of making foamed components of the invention, in the form of tablets, beads or particles

Apparatus: Microbalance, graduated 100ml flask, Kenwood "Chef" food processor with provided whisk and mixing bowl, glass or plastic moulds, spatula.
Chemicals: Poly (vinyl alcohol) (Aldrich chemicals, molecular weight Mw= 30-70k), Glycerol (99 %, Aldrich chemicals), Citric Acid (Aldrich, Citric Acid, USP Anhydrous), distilled water, dry ice (or solid phase CO_2), thermally insulated box.
Procedure

1. Weigh 50 ±0.2 grams of PVA, 30 ±0.2 grams of glycerol, 50 ±0.2 grams of cyclohexane dimethanol.
2. Mix the PVA, glycerol and Citric acid using the mixer set a low speed (mark 2; low sheer).
3. Add 50 ± 1ml of water gradually to the dry mix maintaining the mechanical mix for 2 minutes. A smooth gel should be obtained.
4. Increase the mix speed high sheer to the maximum setting (mark 8). Add 10-20 ml of water until a PVA foam is forming. Maintain high shear mixing for 3 minutes.
5. The active ingredients, for example from 2-10 gram of enzyme, are progressively added to the foam under a maintained mechanical mixing so that a uniform active foam is obtained.
6. Stop mixing. Spread the PVA foam in moulds avoiding any collapsing of the structure.
7. Place the filled moulds in a thermally insulated box 1/3 filled with dry ice. Leave to freeze for 5 hours.
8. Quickly place frozen samples in a vacuum freeze-dryer (Edward XX) for 24 hours.
9. Remove dried sample from moulds.

Any active ingredient can be added in step 5, at any level, normally up to about 50 grams, for example fabric softeners, bleaching species, nonionic surfactants.

Any dissolution aid can be added in step 5, at any level, normally up to about 50 grams, for example cyclohexane dimethanol, or sodium toluene sulfonate.

Example 3

A process of making foam components of the invention, in the form of tablets, beads or particles

Apparatus: as described in the above example

Chemicals: Poly (vinyl alcohol) (Aldrich chemicals, molecular weight Mw= 30-70k), Glycerol (99 %, Aldrich chemicals), Sodium carbonate (Aldrich, Anhydrous), Dodecyl Sulphate surfactant (Aldrich), distilled water, Petri dish (diameter 90 mm), Oven (set at 45 °C ±2°C)

Process:

1. Weigh 50 ±0.2 grams of PVA, 30 ±0.2 grams of glycerol, 20 ±0.2 grams of cyclohexane dimethanol, 20 ±0.2 grams of sodium carbonate, and 2 ±0.2 grams of dodecyl sulphate.
2. Mix the PVA, glycerol, citric acid and dodecyl sulphate using the mixer set a low speed (mark 2).
3. Add 50 ± 1ml of water gradually to the dry mix maintaining the mechanical mix for 2 minutes. A smooth gel should be obtained.
4. Add the active ingredient, for example 5 gram enzyme, and sodium carbonate and mix vigorously for 30 second until a fully expanded foam is obtained
5. Spread the foam in petri dish in a uniform 1cm thick layer
6. Place petri dish in 40 °C oven for 24 hours.
7. Remove the dried foam film from mould.

Any active ingredient can be added in step 4, at any level, normally up to about 50 grams, for example fabric softeners, bleaching species, nonionic surfactants.

Any dissolution aid can be added in step 1 at any level, normally up to about 50 grams, for example SDS, STS, SXS, SCS, CHDM.

This was repeated by using 55wt% polycarboxylic acid polymer, 20wt% anhydrous sodium carbonate and 25wt% enzyme, softening clay etc.; and repeated by using 45wt%

polycarboxylic acid polymer, 15wt% polyethylene glycol, 20wt% anhydrous sodium carbonate and 20wt% enzyme, softening clay etc.